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**HYDRATE PRECIPITATION AND OXALATE REMOVAL**

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(57)

In a Bayer process for the recovery of alumina from bauxite ore, the bauxite is digested in a caustic solution and, after separation from red mud and other insoluble impurities, the green liquor passes through a precipitation circuit (10). Co-precipitation of sodium oxalate and aluminium hydroxide occurs in precipitation circuit (10), the sodium oxalate being precipitated as coarse clusters and/or spherulites by altering process conditions in precipitation and/or adding a crystal growth modifying reagent. Coarse sodium oxalate crystals pass with the underflow from the final precipitator stage to a product classification facility (20), together with the coarser hydrate crystals. Few sodium oxalate crystals pass with the overflow to the seed thickeners (10), and hence little or no seed washing is required in the seed deliquoring and re-slurry facility (14). The coarse oxalate is screened from the product hydrate in hydrate screening facility (24), and the bulk of the product hydrate proceeds to the calcination facility (22). In general, the oxalate clusters are larger than the product hydrate and therefore most of the product hydrate slurry passes through the screen leaving only deliquored coarse oxalate and some coarse hydrate. The deliquored coarse oxalate and coarse hydrate is fed into an oxalate dissolution and hydrate recovery facility (28) in which the sodium oxalate spherulites are dissolved. Following dissolution the

HYDRATE PRECIPITATION AND OXALATE REMOVAL

The present invention relates to a process and system for the removal of sodium oxalate from a Bayer process liquor in an alumina refinery.

5        Precipitation technology in use at many alumina refineries results in the co-precipitation of aluminium hydroxide (hydrate) and sodium oxalate. The sodium oxalate precipitate is typically in the form of fine needles that remain with the finer hydrate and report to the hydrate seed classification and filtration facilities of the alumina refinery. One method of removing sodium oxalate from a precipitation circuit is to wash fine seed hydrate and send 10      the wash water to an oxalate removal facility.

15      Sodium oxalate needles in precipitation contribute to excessive hydrate fines and adversely affect the classification of fine and coarse hydrate in the precipitation and classification circuits. These effects are normally controlled by increasing precipitator temperatures but unfortunately this has an adverse effect on plant hydrate 20      yield.

25      The process according to the invention involves encouraging the growth of sodium oxalate crystals as coarse clusters or with a spherulitic morphology (oxalate balls) as distinct from needles. An object of this modification to the crystal morphology is to improve both the hydrate precipitation and classification processes, and to enable sodium oxalate to be more efficiently removed from the process liquor.

30      The formation of oxalate balls and coarse clusters of oxalate crystals is observed from time to time in most alumina refineries. The occurrence of balls normally creates a product quality problem as the oxalate balls tend to report to the product hydrate filters and are slow to dissolve. A typical result is difficulty in achieving target product soda 35      levels. However, the deliberate modification of the crystal morphology of sodium oxalate to assist in its removal has

previously been proposed. For example, in Australian Patent No. 607014 (24528/88), by C.S.I.R.O. and ALCOA of Australia Limited, a process for removing sodium oxalate from a spent Bayer liquor is described, which involves forming spherulitic sodium oxalate crystals in the spent Bayer liquor and subsequently removing the spherulitic sodium oxalate crystals by filtration or by adding a thickener and settling.

US Patent No 4,608,237, by NALCO CHEMICAL COMPANY describes the use of a crystal growth modifier which is added to the green liquor immediately prior to the precipitation stage. A high molecular weight polyacrylic acid is added to the pregnant Bayer process liquor to modify the growth of sodium oxalate crystals to form large clusters or coarser crystals which are claimed to be innocuous with respect to the co-precipitation of alumina trihydrate (hydrate). According to US 4,608,237, hydrate precipitation using a crystal growth modifier produces a product which is coarser, stronger, contains reduced soda levels, and leads to increased hydrate yield and improved oxalate removal during the alumina washing and filtration steps.

At the applicant's refinery the deliberate creation of coarse oxalate crystals as a more efficient method of removing sodium oxalate from the process liquor has previously been explored. However, this was not considered feasible due to the unavailability of an effective technique for separating the coarse oxalate crystals from the product hydrate. One proposed technique involves screening the coarse oxalate crystals from the product hydrate. However, most precipitation circuits currently produce some relatively coarse hydrate precipitate. The possibility of using this technology has been regarded as impractical up until now due to the production losses that would result from removing significant tonnages of coarse hydrate along with the coarse sodium oxalate during screening.

In applicant's co-pending Australian patent application No. 73182/91 a method and system of biological degradation of sodium oxalate is described. The disclosure of AU 73182/91 is incorporated herein by reference. The

present invention was developed by combining the two technologies of coarse oxalate formation and biological oxalate degradation to overcome the adverse effects of sodium oxalate needles.

5 According to one aspect of the present invention there is provided a process for the removal of sodium oxalate from a Bayer process liquor, the removal process including the following steps:

10 facilitating the growth of coarse clusters and/or spherulites of sodium oxalate in the Bayer process liquor during aluminium hydroxide precipitation;

removing the coarse oxalate and product size aluminium hydroxide together following co-precipitation;

15 separating the coarse oxalate from the bulk of the product aluminium hydroxide;

dissolving the coarse oxalate and recovering any remaining aluminium hydroxide; and,

20 biologically degrading the dissolved sodium oxalate in a biological reactor containing an aqueous solution in which at least one oxalate-degrading aerobic micro-organism is present.

25 Advantageously said step of separating the coarse sodium oxalate comprises a step of screening the coarse sodium oxalate and any coarse aluminium hydroxide from the bulk of finer aluminium hydroxide product. Preferably said step of screening also involves de-liquoring the coarse sodium oxalate and coarse aluminium hydroxide.

30 Typically said step of facilitating the growth of coarse clusters and spherulites of sodium oxalate comprises altering process conditions in precipitation and/or adding a crystal growth modifying reagent to the process liquor prior to and/or during its passage through a precipitation circuit. Advantageously said crystal growth modifying reagent also assists in the agglomeration and strengthening of aluminium hydroxide precipitate.

35 Typically said step of removing the coarse sodium oxalate and product size aluminium hydroxide precipitate comprises classifying the precipitate and returning at least

some finer aluminium hydroxide to the precipitation stage.

According to another aspect of the present invention there is provided a system for the removal of sodium oxalate from a Bayer process liquor, the system comprising:

5 a precipitation circuit for co-precipitation of sodium oxalate and aluminium hydroxide and wherein the sodium oxalate is precipitated as coarse clusters and/or spherulites;

10 means for removing the coarse sodium oxalate and product size aluminium hydroxide from the precipitation circuit;

means for separating the coarse sodium oxalate from the bulk of the aluminium hydroxide;

15 means for dissolving the coarse sodium oxalate and recovering any remaining aluminium hydroxide; and,

20 a biological reactor into which the dissolved sodium oxalate is introduced, the biological reactor containing an aqueous solution in which at least one oxalate-degrading aerobic micro-organism is present for biologically degrading the sodium oxalate.

In one embodiment said means for separating the coarse sodium oxalate from the bulk of the aluminium hydroxide comprises a screening facility for screening the coarse sodium oxalate and any coarse aluminium hydroxide from the bulk of finer aluminium hydroxide product.

30 In order to facilitate a more thorough understanding of the nature of the invention a preferred embodiment of the process and system for the removal of sodium oxalate according to the invention will now be described, by way of example only, with reference to the accompanying drawings in which;

35 Figure 1 is a schematic block flow diagram of the hydrate precipitation, seed preparation, oxalate removal and calcination facilities of a typical alumina refinery; and,

Figure 2 is a schematic block flow diagram of a preferred embodiment of the invention applied to the alumina refinery illustrated in Figure 1.

In the Bayer process for the recovery of alumina from bauxite ore, the bauxite is digested in a caustic solution and, after separation from red mud and other insoluble impurities, the pregnant or green liquor passes through a precipitation circuit. In a typical hydrate precipitation system as illustrated in Figure 1, precipitation is conducted progressively in a precipitation circuit 10 comprising a series of precipitator tanks. The hot pregnant liquor is cooled prior to entry into the first stage of the precipitation circuit, typically within the range 67° to 88° C. Seed crystals of aluminium hydroxide are added to the hot pregnant liquor which is allowed to slowly cool naturally, or more advantageously the liquor may pass through one or more heat interchange stages to more efficiently precipitate aluminium hydroxide.

Simultaneously with the precipitation of aluminium hydroxide, in the cooler regions of the precipitation circuit, sodium oxalate crystals also form. In the final stages of the precipitation process the coarse aluminium hydroxide crystals are separated from the remaining fine hydrate and liquor by a classification facility 20, coarser crystals being removed with the underflow and finer crystals being removed with the overflow. The coarse product hydrate then passes through a washing and calcination facility 22 to produce the product alumina. The finer crystals from the product classification 20 circuit may be returned to the precipitation circuit or further classified and recycled as seed after passing through seed thickeners 12 and a seed preparation facility 14 where the seed may be deliquored, washed and reslurried. Wash water carrying sodium oxalate can then be passed to an oxalate removal facility 16.

A substantial volume of wash water is required to wash the seed hydrate in seed preparation facility 14 free of sodium oxalate. In the oxalate removal facility 16 the sodium oxalate is crystallised from the wash water. The resulting slurry is filtered and the sodium oxalate filter cake disposed of elsewhere. The filtrate from the oxalate removal facility 16 is returned together with spent liquor

from the seed thickeners 12 and seed deliquoring facility 14 to an evaporator 18, and the liquor is then recycled to the bauxite digestion facility.

In Figure 2 a preferred embodiment of the process for controlling the adverse effects of sodium oxalate needles in an alumina refinery is shown. The aluminium hydroxide precipitation circuit and the removal of sodium oxalate from a Bayer process liquor is illustrated in schematic block flow diagram form applied to the same precipitation system described above and illustrated in Figure 1. The same reference numerals are used in Figure 2 as in Figure 1 to identify the common parts of the system. Shaded blocks in Figure 2 represent facilities existing in the system described in Figure 1. It can therefore be seen that the implementation of the new system requires minimal capital expenditure.

In one example of a hydrate precipitation circuit 10, the facility comprises an agglomeration stage followed by two growth stages with interstage cooling between each stage. Careful control of the temperature profile through the precipitation circuit is required in order to optimise both the circuit's aluminium hydroxide yield and the particle size of the product. The particle size distribution of the precipitated aluminium hydroxide is influenced not only by the temperature profile, but also by the seed charge, seed crystal surface area, the presence of sodium oxalate needles, liquor flow, degree of agitation and the presence of impurities in the process liquor such as humates that influence the hydrate crystallisation.

In the above example, the pregnant liquor is typically cooled to approximately 80 - 84°C prior to entry into the first agglomeration stage of the precipitation circuit 10. It then undergoes interstage cooling to approximately 70 - 74°C prior to the liquor passing to the first of two growth stages. After this first growth stage the liquor again passes through an interstage cooling circuit where the temperature is lowered to approximately 64°C. More advantageously the liquor should be cooled to 68-70° for the

first growth stage and 55-58°C or lower in the second growth stage to maximise alumina supersaturation and obtain the resulting production benefits. However in the current system it is usually not possible to lower the temperatures to these levels due to the adverse effects of increased quantities of co-precipitated sodium oxalate needles.

According to the present invention, the growth of sodium oxalate as coarse clusters and in a spherulitic morphology in the hydrate precipitation circuit is facilitated in order to minimise the adverse effects of sodium oxalate needles on the precipitation and classification of aluminium hydroxide. The presence of sodium oxalate as coarse clusters and in a spherulitic morphology will also advantageously aid in the separation and subsequent removal of sodium oxalate from the Bayer liquor circuit.

Typically the growth of sodium oxalate in a spherulitic form and as coarse clusters will be as a result of precipitation process changes and/or the use of chemical reagents that can modify the crystal morphology of sodium oxalate as it crystallises from the process liquor. The addition of a crystal growth modifying reagent to the pregnant liquor can be prior to the first stage of a precipitation circuit 10 and/or during any subsequent stage of the precipitation process. The point chosen for the addition of a crystal morphology modifying reagent is chosen with due consideration of the sodium oxalate saturation in the liquor and the concentration of other compounds in the process liquor such as humates.

Any suitable crystal growth modifying agent may be employed in the present invention, for facilitating the growth of coarse sodium oxalate crystals during hydrate precipitation. Preferably the crystal growth modifying reagent also facilitates the agglomeration and growth of hydrate crystals. Suitable crystal growth modifying reagents are described, for example, in Australian Patent No. 607014 (24528/88) and US 4,608,237 referred to above. Other crystal growth modifying reagents are described in Australian Patent

Application No. 5955/91 by NALCO AUSTRALIA PTY. LTD., US 4,737,352 by NALCO CHEMICAL COMPANY and US 5,041,269 by ALLIED COLLOIDS LTD.. Suitable crystal growth modifying reagents include, but are not limited to, the following:

5 polysaccharides; polysaccharide graft copolymers; polyacrylate and polyacrylamide polymers and their copolymers; humates; polyacrylic acids; fatty acids and fatty alcohols alone or combined with a solvent or dispersant; glycol ethers, etc.

10 The use of a crystal growth modifying reagent has the double benefit of producing coarser, stronger hydrate crystals as well as coarse and/or spherulitic sodium oxalate crystals in the later stages of the precipitation circuit 10. In the system of Figure 2, the coarse sodium oxalate crystals  
15 or oxalate balls pass with the underflow from the final precipitator stage to the product classification facility 20, together with the coarser hydrate crystals. Few sodium oxalate crystals pass with the overflow to the seed thickeners 12, and hence little or no seed washing is required in the seed deliquoring and re-slurry facility 14. Seed crystals of hydrate are recycled into the first stage of  
20 the precipitation circuit 10 as before.

The use of a crystal growth modifying reagent both as an agglomeration aid and for controlling the precipitation  
25 of sodium oxalate crystals in the form of coarse aggregates or balls, has significant effects on the precipitation circuit 10. The elimination of the opportunity for oxalate needles to create excessive hydrate nuclei in precipitation and the improvement in the classification of hydrate in classification equipment allows precipitation temperatures to be lowered with the resulting significant benefits in hydrate precipitation yield being realised. Since the sodium oxalate  
30 is now reporting with the coarse aluminium hydroxide a significantly reduced quantity of solid phase sodium oxalate will be present in the fine seed hydrate and little or no washing of the seed will be required. Equipment in the seed preparation facility 14 will generally only be required to deliquor the seed and a large quantity of water (condensate)

will now be available for use elsewhere in the refinery, for example, to recover soda from bauxite residue. A significant benefit to the productivity of an alumina refinery using this process results from the reduced dilution of the liquor returning to the digestion facility with this wash water no longer present. With a higher liquor to digestion concentration more bauxite can be processed.

Unlike the system illustrated in Figure 1, the coarse underflow of the product hydrate classification facility 20 in the system of Figure 2 will now contain a significant quantity of sodium oxalate present as coarse clusters and spherulites (balls). If this coarse oxalate was allowed to proceed to the product hydrate washing and calcination facility 22 without further processing the sodium oxalate would not be removed from the liquor circuit. In fact some would be retained with the alumina resulting in an undesirably high soda content in the product alumina. It has been found that the coarse oxalate can be successfully screened from the product hydrate using curved vibratory screens having a mesh size of, for example, 0.5 mm. In general, the oxalate clusters and balls are larger than the product hydrate and therefore most of the product hydrate slurry passes through the screen leaving only deliquored coarse oxalate and some coarse hydrate. As noted above, in many alumina refineries the coarseness of the product hydrate would result in many tonnes of hydrate reporting to the screen over-size. This would be an unacceptable production loss, however by passing the screen over-size material through an oxalate dissolution and hydrate recovery step this production loss can be eliminated. As any hydrate retained with the coarse oxalate will be recovered in the hydrate recovery step, the screen mesh size can be made smaller if some of the oxalate is found to be passing through the screens and contaminating the alumina.

The bulk of the product hydrate passes through the screens of the hydrate screening facility 24 and proceeds to the calcination facility 22 as before.

The oxalate dissolution and hydrate recovery facility 28 typically comprises highly agitated tank(s) necessary to suspend the coarse oxalate/hydrate solids in process water/condensate/bioreactor effluent mixture. Any one or combination of these fluids may be used to dissolve the sodium oxalate spherulites. Following dissolution the hydrate is recovered by settling or filtration, for example by using redundant filters from the seed deliquoring/washing facility 14 or the oxalate removal facility 16. Advantageously two agitation tanks in series, using an air lift or mechanical agitator, can be used to dissolve the coarse sodium oxalate. The product hydrate can be settled or filtered from the oxalate solution. Examples of filters include, a (vacuum) disk filter, drum filter or horizontal pan filter. The recovered hydrate can be returned to the product hydrate circuit for calcination in facility 22. The clarified liquor from the hydrate recovery facility 28, comprising sodium oxalate solution with any impurities of the process water/condensate/bioreactor effluent used for the oxalate dissolution, passes to a biological reactor 30 for the biological degradation of the sodium oxalate.

If it is desirable to remove the coarse hydrate from the product alumina, then a hydrate/oxalate grinding step prior to dissolution could be installed. The hydrate/oxalate grinding facility 26 may comprise, for example, a small ball mill for grinding the coarse sodium oxalate and coarse hydrate into fines. The grinding facility will also assist in the dissolution of the coarse sodium oxalate clusters and spherulites. If a grinding facility is installed then the hydrate separated from the oxalate solution may be returned to either the product hydrate washing and calcination facility 22 or to the seed deliquoring and reslurry facility 14.

The biological reactor 30 is preferably similar to the bioreactor described in applicant's co-pending Australian patent application No. 73182/91 which employs an alkalophilic oxalate-degrading aerobic microorganism in aqueous solution. The biological reactor 30 comprises a vessel for containing

the aqueous solution and an input for receiving the dissolved sodium oxalate from the oxalate dissolution facility 28. The biological reactor can be operated, for example, automatically as a sequenced batch reactor, or as a continuous feed process. The aqueous solution is aerated and biological degradation of the sodium oxalate in the alkaline solution occurs, with sodium carbonate and bicarbonate formed as end products in the effluent. The effluent passes to a low concentration causticiser reaction tank 32. The sodium carbonate and bicarbonate in the effluent are typically converted to useful sodium hydroxide which can be recycled in the Bayer process. The low concentration and the absence of lime-consuming alumina in the causticiser 32 result in a high causticisation efficiency. The alkalophilic oxalate-degrading aerobic microorganism employed in the biological reactor 30 is typically a Bacillus species of the kind described in AU 73182/91.

Thus, the above-noted problem of significant tonnages of coarse hydrate being screened along with the coarse oxalate is overcome in the above process by passing the coarse product hydrate with coarse oxalate through an oxalate dissolution and hydrate recovery facility. The technology of oxalate dissolution and hydrate recovery would not be an acceptable solution to overcoming the above-noted production loss without the provision of the biological reactor. The dissolved oxalate filtrate from the hydrate recovery facility can be biologically degraded in the biological reactor 30, whereas it could not otherwise safely be disposed of in a cost-effective manner. Hence the combination of coarse oxalate formation technology with biological oxalate degradation provides a new and improved process for the elimination of the precipitation and classification problems caused by sodium oxalate needles while removing sodium oxalate from the Bayer process and recovering soda from it.

Some of the advantages of the above described process for the removal of sodium oxalate from the Bayer process liquor may be summarised as follows:

(1) Control of the seed balance in the precipitation circuit due to the elimination of the fines generation and classification problems caused by sodium oxalate needles.

5 (2) Lower precipitator temperatures will be possible resulting in a higher yield of aluminium hydroxide from the liquor in precipitation.

10 (3) Oxalate crystallisation in coarse clusters or with spherulitic morphology and the separation of the coarse oxalate by screening reduces or eliminates the need for seed washing saving substantial quantities of condensate. This condensate can be used to recover soda values from bauxite residue in the red area of the alumina refinery and will also result in higher liquor to digestion concentrations allowing more bauxite to be digested.

15 (4) Equipment such as crystallisers, filters and pumps in the oxalate removal facility 16 will no longer be required. The oxalate removal inefficiencies in both the seed washing facility 14 and oxalate removal facility 16 are eliminated.

20 (5) The soda losses with oxalate disposal and the cost of operating disposal sites are eliminated.

25 Now that a preferred embodiment of the process and system for the removal of sodium oxalate from a Bayer process liquor has been described in detail, numerous variations and modifications will suggest themselves to persons skilled in the relevant arts. For example, the growth of coarse sodium oxalate during hydrate precipitation may be facilitated by other means, in addition to or as alternatives to the use of a crystal growth modifying reagent. For example, coarse

sodium oxalate crystals can also be formed by careful control of precipitation temperature, concentration of dissolved sodium oxalate and the addition of nucleation aids. Naturally occurring organic substances in the Bayer process liquor, such as humates and fulvates may also act as crystal growth modifiers. Obviously, any suitable facility for the separation of the coarse sodium oxalate from the product hydrate may be employed, other than the described screening facility. Furthermore, the biological degradation of the sodium oxalate can be effected in any suitable bioreactor and is not limited to the method and system disclosed in commonly owned AU 73182/91. The system and method of AU 73182/91 is particularly advantageous because the alkalophilic oxalate-degrading aerobic microorganism described therein can cope with the caustic present with the dissolved oxalate. However, in some refineries it may be feasible to operate at a lower pH level and an alternative microorganism can be employed in the bioreactor.

All such variations and modifications are to be considered within the scope of the present invention, the nature of which is to be determined from the foregoing description and the appended claims.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A process for the removal of sodium oxalate from a Bayer process liquor, the removal process including the following steps:

5 facilitating the growth of coarse clusters and/or spherulites of sodium oxalate in the Bayer process liquor during aluminium hydroxide precipitation;

removing the coarse oxalate and product size aluminium hydroxide together following co-precipitation;

10 separating the coarse oxalate from the bulk of the product aluminium hydroxide;

dissolving the coarse oxalate and recovering any remaining aluminium hydroxide; and,

15 biologically degrading the dissolved sodium oxalate in a biological reactor containing an aqueous solution in which at least one oxalate-degrading aerobic micro-organism is present.

2. A process for the removal of sodium oxalate as defined in claim 1, wherein said step of separating the coarse sodium oxalate comprises a step of screening the coarse sodium oxalate and any coarse aluminium hydroxide from the bulk of finer aluminium hydroxide product.

25 3. A process for the removal of sodium oxalate as defined in claim 2, wherein said step of screening also involves de-liquoring the coarse sodium oxalate and coarse aluminium hydroxide.

4. A process for the removal of sodium oxalate as defined in any one of claims 1 to 3, wherein said step of facilitating the growth of coarse clusters and spherulites of sodium oxalate comprises altering process conditions in precipitation and/or adding a crystal growth modifying reagent to the process liquor prior to and/or during its passage through a precipitation circuit.

5. A process for the removal of sodium oxalate as defined in claim 4, wherein said crystal growth modifying reagent also assists in the agglomeration and strengthening of aluminium hydroxide precipitate.

5 6. A process for the removal of sodium oxalate as defined in claim 5, wherein said step of removing the coarse sodium oxalate and product size aluminium hydroxide precipitate comprises classifying the precipitate and returning at least some finer aluminium hydroxide to the 10 precipitation stage.

7. A system for the removal of sodium oxalate from a Bayer process liquor, the system comprising:

15 a precipitation circuit for co-precipitation of sodium oxalate and aluminium hydroxide and wherein the sodium oxalate is precipitated as coarse clusters and/or spherulites;

means for removing the coarse sodium oxalate and product size aluminium hydroxide from the precipitation circuit;

20 means for separating the coarse sodium oxalate from the bulk of the aluminium hydroxide;

means for dissolving the coarse sodium oxalate and recovering any remaining aluminium hydroxide; and,

25 a biological reactor into which the dissolved sodium oxalate is introduced, the biological reactor containing an aqueous solution in which at least one oxalate-degrading aerobic micro-organism is present for biologically degrading the sodium oxalate.

8. A system for the removal of sodium oxalate as 30 defined in claim 7, wherein said means for separating the coarse sodium oxalate from the bulk of the aluminium hydroxide comprises a screening facility for screening the coarse sodium oxalate and any coarse aluminium hydroxide from the bulk of finer aluminium hydroxide product.

9. A process for the removal of sodium oxalate substantially as herein described with reference to and as illustrated in Figures 2 and 3 of the accompanying drawings.

10. A system for the removal of sodium oxalate substantially as herein described with reference to and as illustrated in Figures 2 and 3 of the accompanying drawings.  
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Dated this 17th day of November 1993.

WORSLEY ALUMINA PTY. LTD.  
By Its Patent Attorneys

10 GRIFFITH HACK & CO.  
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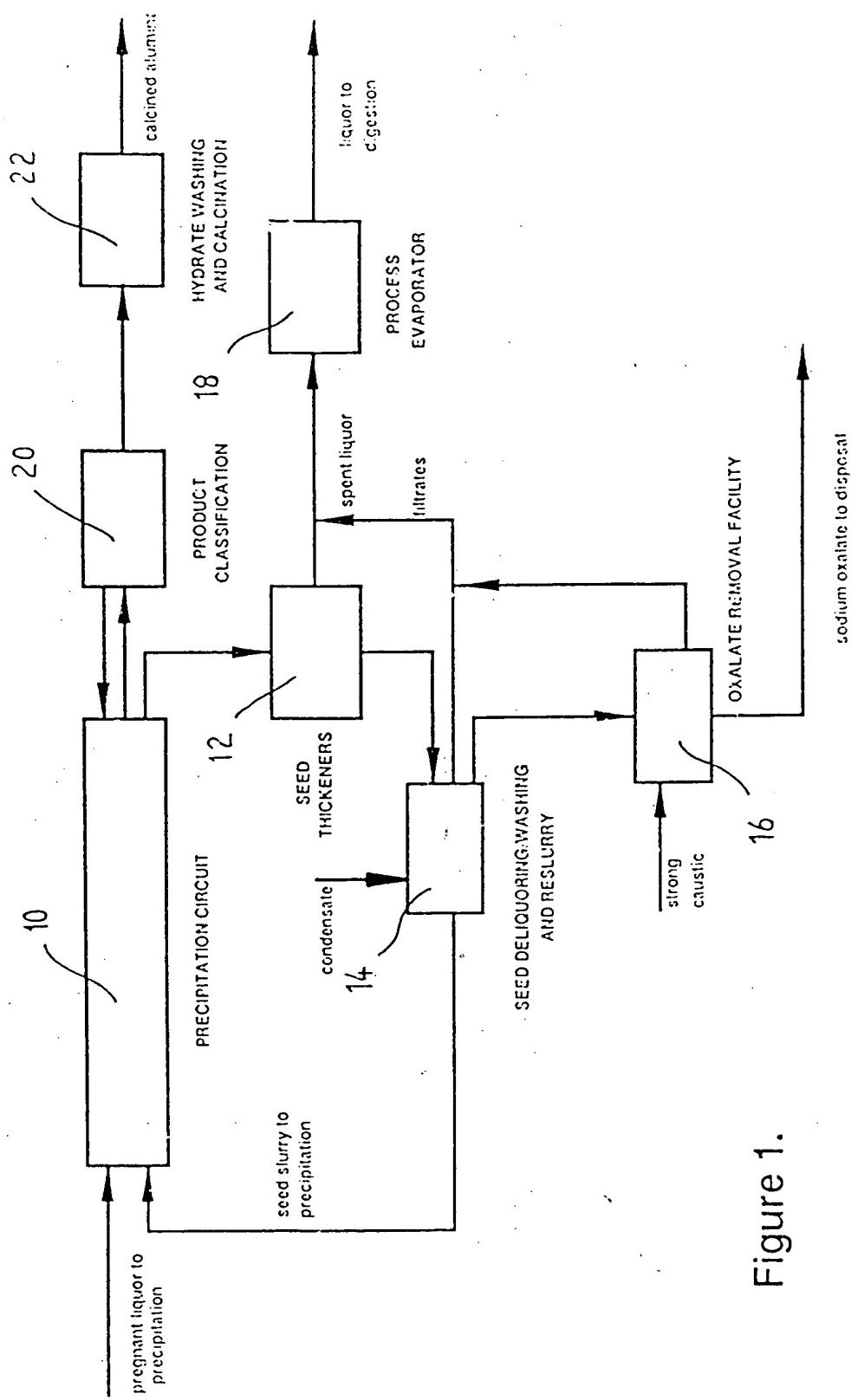


Figure 1.

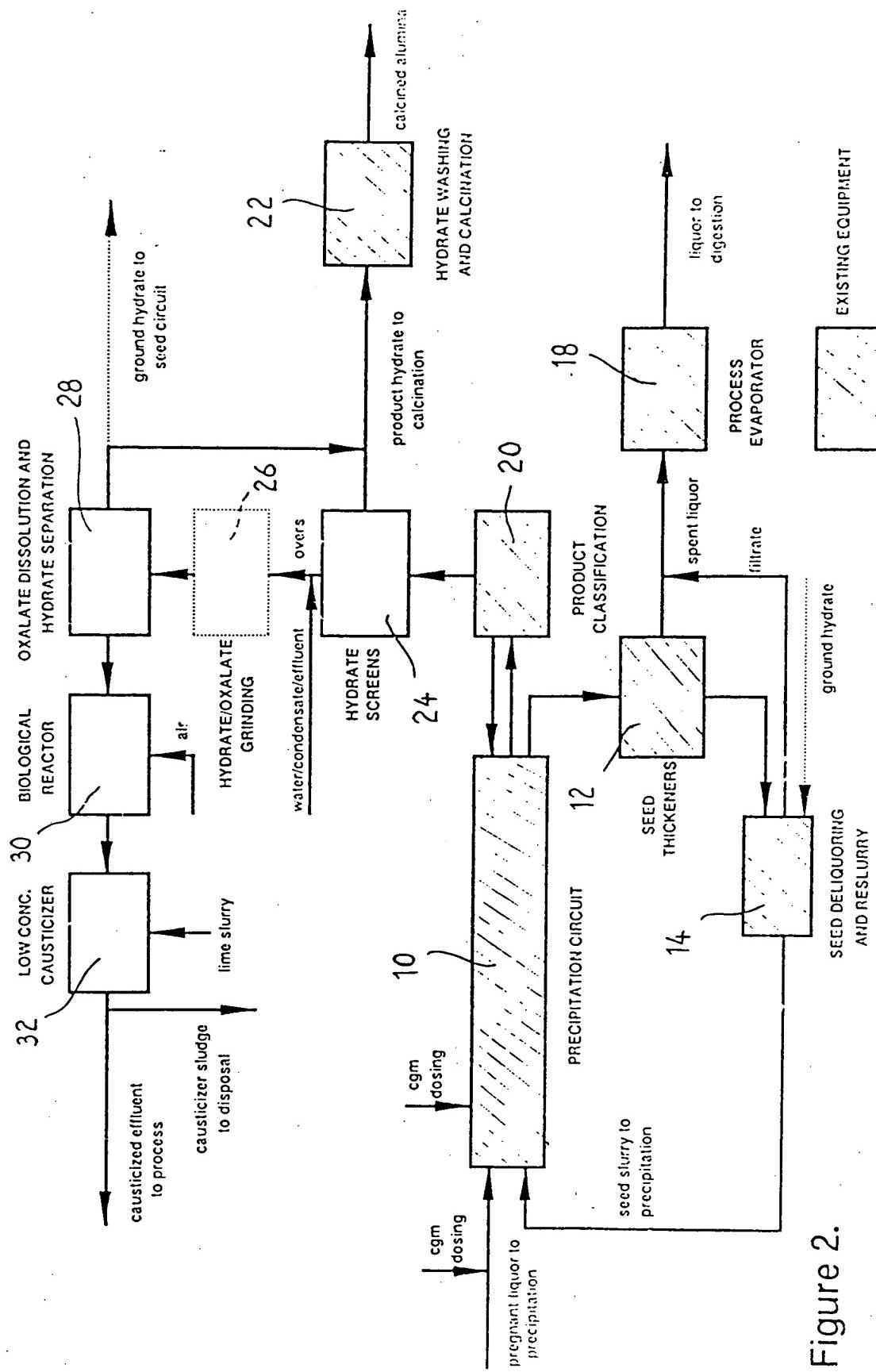


Figure 2.